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Global and local strain rate sensitivity of bimodal Al-laminates produced by accumulative roll bonding

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ABSTRACT

Laminated metallic composites of 5 mm thickness, with a bimodal grain size distribution and alternating layers of commercial purity aluminum (Al99.5) and high purity aluminum (Al99.999) were successfully processed by accumulative roll bonding at room temperature up to 8 cycles. With increasing number of ARB cycles the layers of commercial purity aluminum become ultrafine-grained, whereas the grain size in the high purity aluminum remains clearly above 1 μm. The mechanical properties of the laminates were investigated by compression testing with strain rate jumps and by nanoindentation using continuous stiffness method and nanoindentation strain rate jump tests. Thereby the strain rate sensitivity was determined both globally, proving the bulk laminate material, and locally, proving the single layers of the laminate. It was found that the local strain rate sensitivity of the ultrafine-grained Al99.5-layers is increasing up to 4 ARB cycles, while the local strain rate sensitivity of the coarse grained Al99.999-layers remains rather constant independently of the number of cycles. In contrast to that, the global strain rate sensitivity of the bulk laminate increases up to 8 cycles without saturation. Thermally activated annihilation of dislocations at the grain boundaries and interfaces between the coarse grained and ultrafine-grained layers is proposed in order to interpret the findings.

1. Introduction

Accumulative roll bonding (ARB) [1] is a process of severe plastic deformation which allows the production of ultrafine-grained (UFG) sheet materials with extraordinary mechanical properties. The rather good ductility of these materials [2–5] is closely related to enhanced strain rate sensitivity (SRS), typically found [6–8]. However, the dominating mechanisms leading to the enhanced SRS are not fully clarified until now. Especially bimodal materials which exhibit microstructures with conventionally sized grains (CG) in the micrometer regime, embedded in an ultrafine-grained matrix with grain sizes smaller than 1 μm can provide beneficial mechanical properties, like for instance enhanced fatigue performance [9]. Beyond that, ARB provides the possibility to produce multi-component materials with tailored properties [10] by introducing reinforcements between the sheets [11–13] or by combining different materials [14–16]. The latter allows the combination of heat-treatable and non-heat-treatable alloys of the same type of material. Thereby laminates with a bimodal grain size distribution can be produced, that show alternating CG- and UFG-grains on discrete layers. In this context, first efforts were made by Quadir et al. [17,18], who combined a high-purity Al alloy and a supersaturated Al-0.3 wt.% Sc alloy by ARB. By subsequent annealing, alternating layers of coarse grains and a recovered substructure were produced. Thereby the precipitation of Al_3Sc particles stabilized the latter by boundary pinning. Recently Chekhonin et al. [19,20] have published some results on a laminate composite processed by combining commercially purity aluminum and high-purity aluminum via ARB. They found that in the high-purity aluminum metadynamic recrystallization occurs, while the layers of commercially pure aluminum become ultrafine-grained, leading to a laminated, layerwise bimodal microstructure of the sheet. Thereby the grain growth of the high purity aluminum is limited in normal direction by the layer thickness. They also report that especially the microstructural development in the high purity layers significantly affect the mechanical properties during tensile

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testing. In the present study the same material system with an increased sheet thickness of 5 mm was processed in order to examine the global SRS of the bulk laminate material by compression strain rate jump tests and the local SRS by nanoindentation. In this context Maier et al. [21] have introduced a method for determining the local strain rate sensitivity by nanoindentation strain-rate jump tests. They adapted a standard continuous stiffness method (CSM) to perform several abrupt changes in the applied strain rate at defined indentation depths during one single indentation. It has been shown on nanocrystalline Ni and other homogeneous materials that the SRS determined with this nanoindentation technique matches very well with global data determined by compression tests. Previously reported differences could be explained by artefacts involved when no jump technique is used, see the work by Hay et al. [22]. Consequently, in this work the SRS of the single layers in the laminate could be examined and compared to the global results, which contributes to the ongoing discussion about the deformation mechanisms in UFG materials. The investigations of the mechanical properties were accompanied by microstructural examinations in the SEM.

2. Experimental

Sheet-laminates of 5 mm thickness with alternating layers of commercial purity aluminum Al99.5 and high-purity aluminum Al99.999 (henceforth labeled as Al99.9) where processed by accumulative roll bonding up to 8 cycles (N8). Prior to ARB the Al99.5 sheets where annealed at 500 °C for 1 h, while the Al99.9 sheets were used as-delivered, these conditions are henceforth denoted as N0. After wire brushing, the sheets were stacked and riveted together in order to prevent partitioning during rolling. Finally the sheets were roll bonded without lubrication and with a thickness reduction of 50% in each cycle. For ARB-processing a Carl Wezel four-high rolling mill (type BW 300) with a working roll diameter of 90 mm was used at a rolling speed of 1 m/min. Microstructural investigations were performed using a Zeiss Crossbeam 1540 EsB scanning electron microscope in backscattered electron (BSE) contrast at an acceleration voltage of 12.5 kV and a working distance of 7–8 mm. The mechanical properties were determined by compression testing using an Instron 4505 universal testing machine. Thereby compression tests with strain rate jumps between 10^{-3}, 10^{-4} and 10^{-5} s^{-1} were performed at room temperature along three directions of the sheets, namely the rolling direction (RD), the transverse direction (TD) and the normal direction (ND). The compression samples were cuboid and had an aspect ratio length/width of 1.3. The global strain rate sensitivity was determined by back-extrapolation from a quasi-static stress state within a regime of constant strain rate towards the jump and using the following equation according to Hart et al. [23]:

$$m = \frac{\partial ln\sigma}{\partial ln\dot{\varepsilon}}\Big|_{\varepsilon_{pl}=const.} \quad (1)$$

This procedure was performed in order to minimize effects of the transient areas, which appear due to the change in dislocation structure directly after the strain rate jump, see Ref. [24] for details.

In addition to that nanoindentation experiments were performed using a Nanoindenter XP (MTS Nanoinstruments, Oak Ride, TN, USA) equipped with a three-sided Berkovich pyramid [25]. Thereby CSM with an indentation depth of 2000 nm was applied. Neighboring indents were separated by 70 μm in both directions to avoid any influence of the plastic zone around the indent. Tip shape calibration was done according to the Oliver–Pharr method [26]. The measurements were performed on the rolling plane 30° to ND and over half of the sheet thickness. Moreover nanoindentation strain rate jump tests were performed for determining the local strain rate sensitivity in individual Al99.5 and Al99.9 layers, see Ref. [21] for details of the method. The indentation strain rate was varied between 1×10^{-2}, 5×10^{-2} and 5×10^{-3} s^{-1} within an indentation depth of 2200 nm. The strain rate sensitivity was determined from hardness measurements using the indentation strain rate as defined by Mayo et al. [27]:

$$m = \frac{\partial lnH}{\partial ln\dot{\varepsilon}} \quad (2)$$

The measurements were conducted on the middle layers and the outside layers of the laminate. The results were averaged over three indents where always three strain rate jumps were evaluated.

3. Results

In Fig. 1a and b the microstructures of the initial materials are shown in BSE contrast. Both have rather equiaxed grains which are in the range of 50–200 μm for Al99.5 and 50–100 μm for Al99.9. After 2 cycles (Fig. 1c) the microstructure of the Al99.5-layers already appears to be rather fine grained with a median grain size of around 0.5 μm in ND, while the 99.9-layers are pretty coarse with a median grain size of 46 μm similar to the initial condition. Moreover the grains are only slightly elongated with an aspect ratio of 1.83 (RD/ND), which is in good agreement with results obtained by Kamikawa et al. [28,29], who investigated the microstructure and the mechanical properties of high-purity Al (99.99wt.%) after ARB. They found that the majority of the structure is composed by euqi-axed crystallites, with an aspect ratio of 1.6, surrounded by high-angle boundaries. According to Chekhonin et al. [19] the microstructure of the high purity layers changes during ARB due to deformation and discontinuous recrystallization. After 4 cycles they found a grain size of 37 μm which is limited by the layer thickness only. It has to be noted, that they used 1 mm thick sheets. However, in the present study the layer thickness is much higher, thus the grain size in the Al99.9-layer is still smaller than the layer thickness after four passes (Fig. 1d). Some grains of the high-purity layers reach the dimension of the layer thickness in ND after N6. Due to the structural limitation in ND the aspect ratio increases to 2.89. After N8 cycles the majority of the grains has reached the layer thickness and the aspect ratio has further increased (Fig. 1e and f). In order to clarify more about that issue, a grain size analysis was performed within multiple Al99.5 and Al99.9-layers after N2 to N8 using the line intersection method. In Fig. 2 the cumulative frequency is plotted over the grain size.

It is found that the Al99.5-layers almost exclusively show a grain size smaller than 1 μm in ND, already after N2. Moreover it is found, that also the grain size in the Al99.9-layers is decreasing with the number of ARB-cycles, however clearly remains in the CG-state. As the layer thickness is also decreasing with the number of ARB-cycles, the median grain size within the Al99.9-layers approaches the layer-thickness after N8. For better comparison, the theoretical layer thickness, the measured layers thicknesses, as well as the median grain size within the Al99.5 and the 99.9-layers and the aspect ratios are summarized in Table 1. Initial deviations of the theoretical and the measured layer thicknesses are due to a thickness reduction slightly higher than 50% in the first ARB-cycle. Standard deviations derive from a slightly developed waviness of the layers. Due to the high degree of cold working, grain sizes after N1 could not be determined distinctly and are therefore omitted.

The results from nanoindentation testing using the CSM method are shown in Fig. 3. Thereby hardness profiles along cross sections of the sheets could be determined. In the initial N0 condition the hardness of both materials is around 0.20 GPa. With increasing